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# Drilling Waste Management

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# Drilling Waste Management

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## Drilling Waste Management Technical Document

### Background

This document describes drilling waste management procedures (disposal methods and criteria) developed with input from government and industry representatives on the Drilling Waste Review Committee. It outlines sampling and analytical requirements, data reporting requirements, and criteria for the main disposal options. These requirements and criteria are based on the results of a rigorous sampling and characterization study of 114 drilling sumps which was conducted in 1990/1991.

The goal of all disposal options is to protect the environment and return the disposal site and any affected areas to equivalent land capability. To accomplish this goal, the operator is encouraged to be aware of the site specific reclamation and certification requirements. The following disposal criteria were developed for general use and application throughout the province. Site specific soil conditions may require innovative and/or more rigorous requirements than those identified in this Directive.

Regulatory jurisdictions remain unchanged. Waste disposal on private land will be administered by the appropriate Energy Resources Conservation Board (ERCB) area office (Figure 1). The appropriate public lands management agency will approve waste disposal on public lands (Figure 2).

### Sampling Requirements

All liquids and solids produced from the drilling operation must be sampled and analyzed to determine their characteristics. It must be recognized that in characterizing a sump the analytical work and interpretation of results are only as good as the samples obtained, therefore care and caution must be employed during the sampling process. The samples must be representative of the entire sump contents and must be collected in a manner that represents the plan for disposal. For example, for sumps with recoverable hydrocarbon on the surface, the sample collected should include the hydrocarbon material, if the intent is to dispose of the sump liquids without first removing the recoverable hydrocarbon. Conversely, if the intent is to remove the hydrocarbon prior to disposal, then the sample should be collected without the hydrocarbon.

Certain materials (eg. lube oil, camp waste, cement returns, biocides, detergents, etc.) not associated with the drilling procedure can interfere with the drilling waste analyses and loading rate calculations. These materials must not be added to the sump, but disposed of separately.

This section describes the general procedures that should be employed to obtain representative samples for analyses. The outline provided cannot take into consideration all the different site specific conditions that may be encountered during sampling.

## 1. Information Requirements

Background information regarding the contents of the sump sampled, and a description of the location and sampling procedures utilized, is important in the interpretation of the analytical results and development of disposal options. A sampling site description form should be completed for each sump location sampled. The form should document the following:

- name(s) of the sampler(s),
- date and time of sampling,
- legal location of sump (well) site and well licensee,
- sketch of sump(s) denoting dimensions (length, width, depth) and approximate subsample site locations,
- type of containers used,
- general comments regarding appearance of sump and material sampled, i.e. colour, "texture", odour if any, and
- ease or difficulty in obtaining samples with an explanation of problems encountered.

## 2. Sampling Methods

Methods utilized will vary based on size and nature of the sump and its contents. It may be possible to adequately sample very small sumps from the edge. However, this approach would likely not be suitable for sumps where the smallest dimension is in excess of 8 m.

A number of alternatives, including the use of backhoes, cherry pickers and boats, are available for sampling sumps that cannot be adequately sampled by hand from the edge. The equipment and/or approach utilized for sampling a sump will be based on site specific conditions and sampler preference, however the ultimate goal of obtaining representative samples must be achieved.

A variety of suitable sampling tools are available. The tools should be fabricated from materials which minimize the potential for sample contamination.

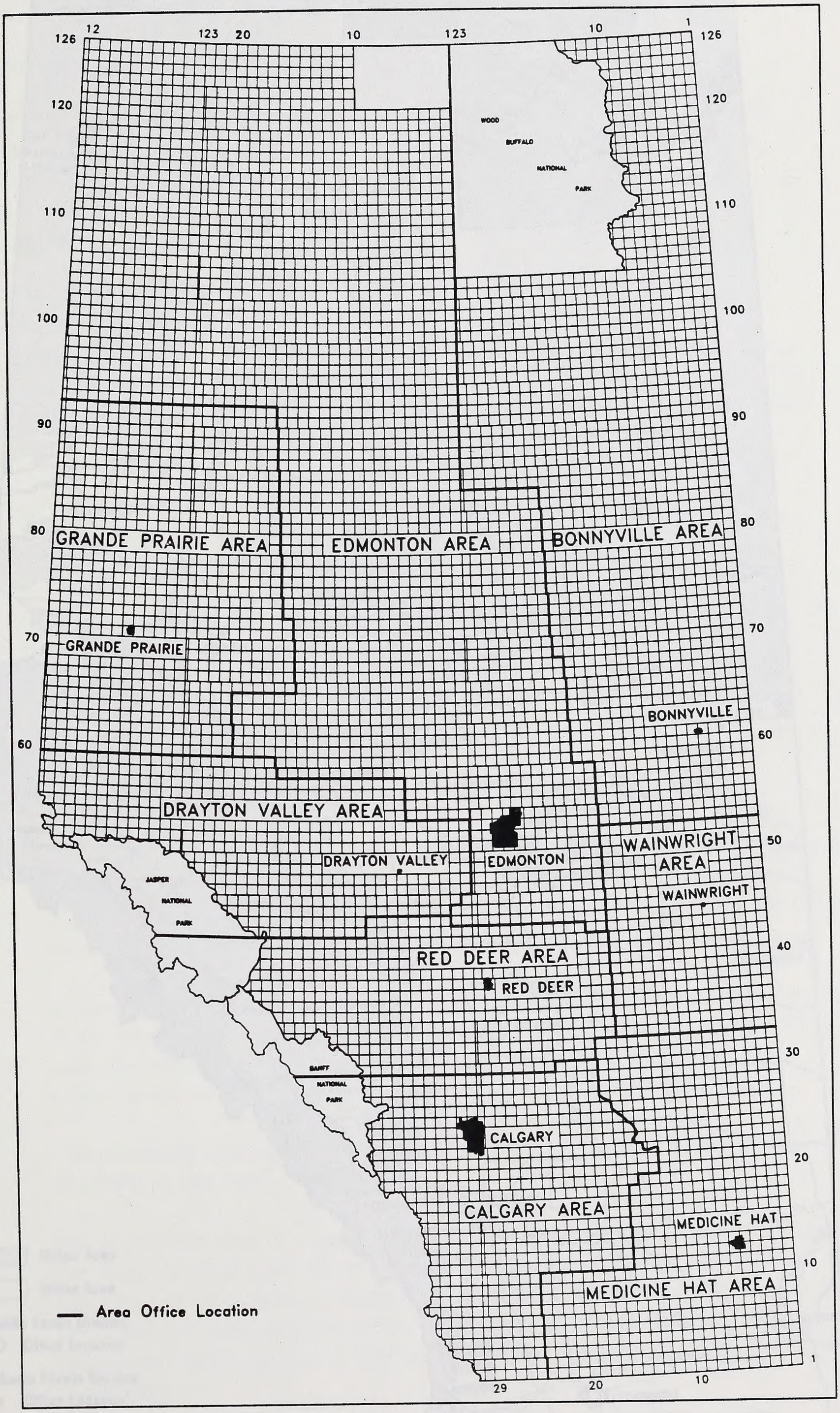

FIGURE 1: AREA OFFICE INSPECTION DISTRICTS

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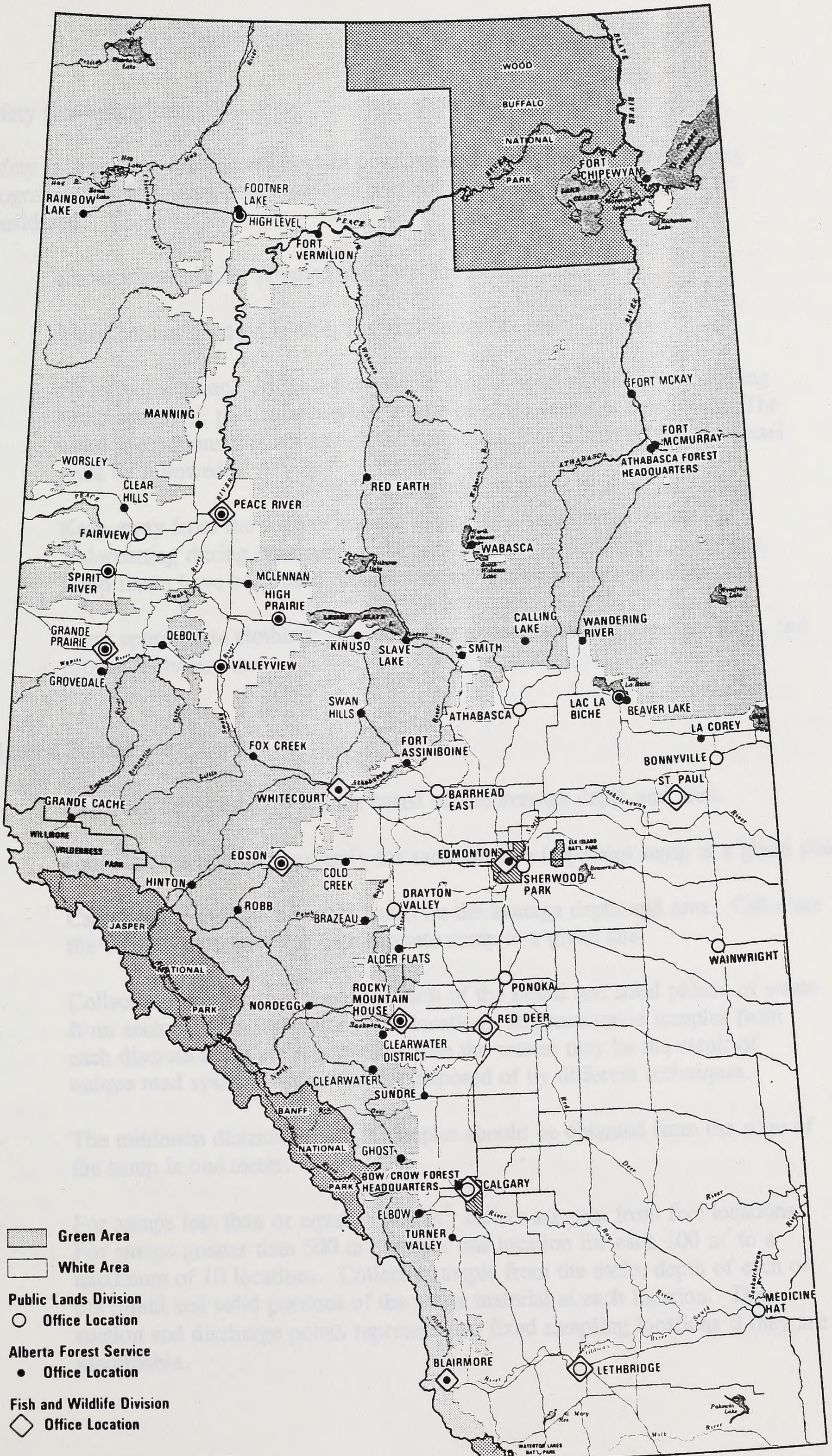


▨ Green Area

☐ White Area

**Public Lands Division**

○ Office Location

**Alberta Forest Service**

● Office Location

**Fish and Wildlife Division**

◇ Office Location

Prepared by Resource Graphics, L.I.S.D., 1991.

## 3. Safety Considerations

Safety is the prime consideration when planning and executing a sump sampling program. The following points address some of the major factors that must be considered.

- Know what is being sampled and what the hazards are.
- Make certain the area is safe, i.e. $H_2S$ detection, etc.
- Never work alone. At least two people should be present/involved during sump sampling, particularly when a boat or other vessel is being used. The usual precautions that are associated with the use of a boat or similar vessel must be followed.
- Keep away from the edge of sumps. They may be unstable because of undercutting during construction. Under wet or icy conditions, or where sump liners have been used, secure footing is a major consideration.
- Wear appropriate clothing; gloves, safety glasses, coveralls, special suits, and proper footwear, etc.

## 4. General Procedures

- Calculate the volume of liquids based on the average depth and area.
- Calculate the volume separately for each discrete (separate) sump at a given site.
- Calculate the volume of solids based on the average depth and area. Calculate the volume separately for each discrete sump at a given site.
- Collect a representative sample of each of the liquid and solid phases of waste from each discrete sump at a given location. Representative samples from each discrete sump are required because the wastes may be the result of unique mud systems which may be disposed of by different techniques.
- The minimum distance at which samples should be obtained from the edge of the sump is one meter.
- For sumps less than or equal to 500 $m^2$, collect samples from five locations. For sumps greater than 500 $m^2$, sample one location for each 100 $m^2$ to a maximum of 10 locations. Collect a sample from the entire depth of each of the liquid and solid portions of the waste material at each location. The suction and discharge points represent two fixed sampling locations if they are identifiable.

- Prepare one composite sample of the liquid phase materials by combining equal volumes of the liquid material collected at each sample location. Composite samples should be collected in a clean container and then transferred to the appropriate sample container as soon as possible for transport. Follow the same procedure for preparing a composite solid phase sample.

- The samples should be placed in containers according to the types of analyses to be conducted. Acid washed glass jars with teflon lids are required for samples which will be analyzed for organic components (oil content) and Microtox® bioassay. Uncoloured plastic containers (bags, bottles) are required for samples which will be analyzed for inorganic components (salts, metals, pH, others). This type of container is required to minimize the potential for sample contamination from coloured plastics.

## 5. Liquids

For sumps with recoverable hydrocarbons on the surface, collect the sample according to the planned disposal procedure. If the hydrocarbon is to be removed prior to disposal, do not include the hydrocarbon layer in the sample. If the hydrocarbon will not be removed prior to disposal, then include the hydrocarbon layer in the sample.

Place portions of the composite liquid sample in containers as follows:

- A minimum of one litre in a uncoloured plastic container.

- One litre in an acid washed glass jar with a teflon lid.

- Keep the samples at or below 4 °C and deliver to the laboratory within 48 hours from the time of sampling. This may necessitate the use of portable coolers and ice in warm weather.

## 6. Solids

Place a minimum of one kg of the composite solid sample in an acid washed glass jar, and one kg of the composite sample in an uncoloured plastic container. If a plastic bag is used, care must be taken to avoid loss of sample (puncture, leakage) and/or obscuring the sample label. Double or triple-bagging is recommended.

## 7. Soil

If either the liquid or solid wastes are to be landspread, the receiving soil must be sampled prior to disposal.

For locations where the waste will be spread on and incorporated into the subsoil, obtain one composite sample comprised of equal amounts of 5 subsamples collected from the 0 - 30 cm depth in the horizon to be used for disposal.

For locations where the waste will be spread on and incorporated in the surface soil or "topsoil", obtain one composite sample comprised of equal amounts of 5 subsamples collected from the 0 - 15 cm depth in the horizon to be used for disposal. At locations where the surface soil or "topsoil" has been stockpiled prior to sampling, subsamples must be obtained to represent the entire stockpile.

Place a minimum of 2 kg of soil in a uncoloured plastic container or bag. Precautions must be taken to avoid loss of sample or obscuring the identification label. Double-bagging is recommended.

## Analytical Requirements

The purpose of the analyses described herein is to obtain the data required to develop waste treatment and/or management plans. If a management plan has already been selected, the data collected will determine if landspreading is feasible (spread rates, area of land needed), or confirm the acceptability of other disposal options.

Note that drilling waste disposal will require knowledge of the resulting waste/soil mix chemistry. This means that calculations of the parameters in the waste/soil mix must be made, or that additional sampling and analysis of the waste/soil mix is required.

Additional information regarding the analytical requirements discussed below is provided in Tables 1, 2, 3 and 4.

- Liquids

  Liquids must be analyzed to determine: pH, specific conductance, total suspended solids, nitrogen (nitrate and ammonia), major ions (Ca, K, Mg, Na, Cl, $SO_4$), and Microtox® bioassay, according to the methods in Table 1. Sodium adsorption ratio (SAR) must also be calculated.

- Solids

  Solids must be analyzed to determine: corrected wet bulk density, pH, electrical conductivity (EC), oil content, water content, major ions (Ca, K, Mg, Na, Cl), and texture according to the methods in Table 2. Sodium adsorption ratio (SAR) must also be calculated.

- Metals

  (Cd, Cr, Cu, Ni, Pb, V, Zn) and boron (B) must be analyzed in the solids if:

  (a) they were added as part of the drilling mud formulation in excess of the amounts specified in Table 4 or,

  (b) the well depth is greater than 2300 m or,

  (c) the waste is to be landspread on an area less than 0.75 ha.

- Soils

  If the waste is to be landspread or mix / bury / covered, analyze soil pH, EC, and major ions (Ca, K, Mg, Na, and Cl) and calculate SAR prior to disposal using the methods provided in Table 3.

- Toxicity Assessment

  A toxicity assessment on the liquids is required for the prediction of potential biological impacts of waste disposal. The bio-luminescent bacteria (Microtox® ) is used in a staged approach (Figure 3) to determine the probable causes of toxicity in the liquids and to assist in developing waste treatment options. Waste materials failing to pass the toxicity test may require extensive analysis to determine the source of the toxicity and/or additional biological testing.

  The bio-luminescent bacteria bioassay is performed on the drilling waste liquids using the method defined by the Energy Resources Conservation Board (see Appendix 1).

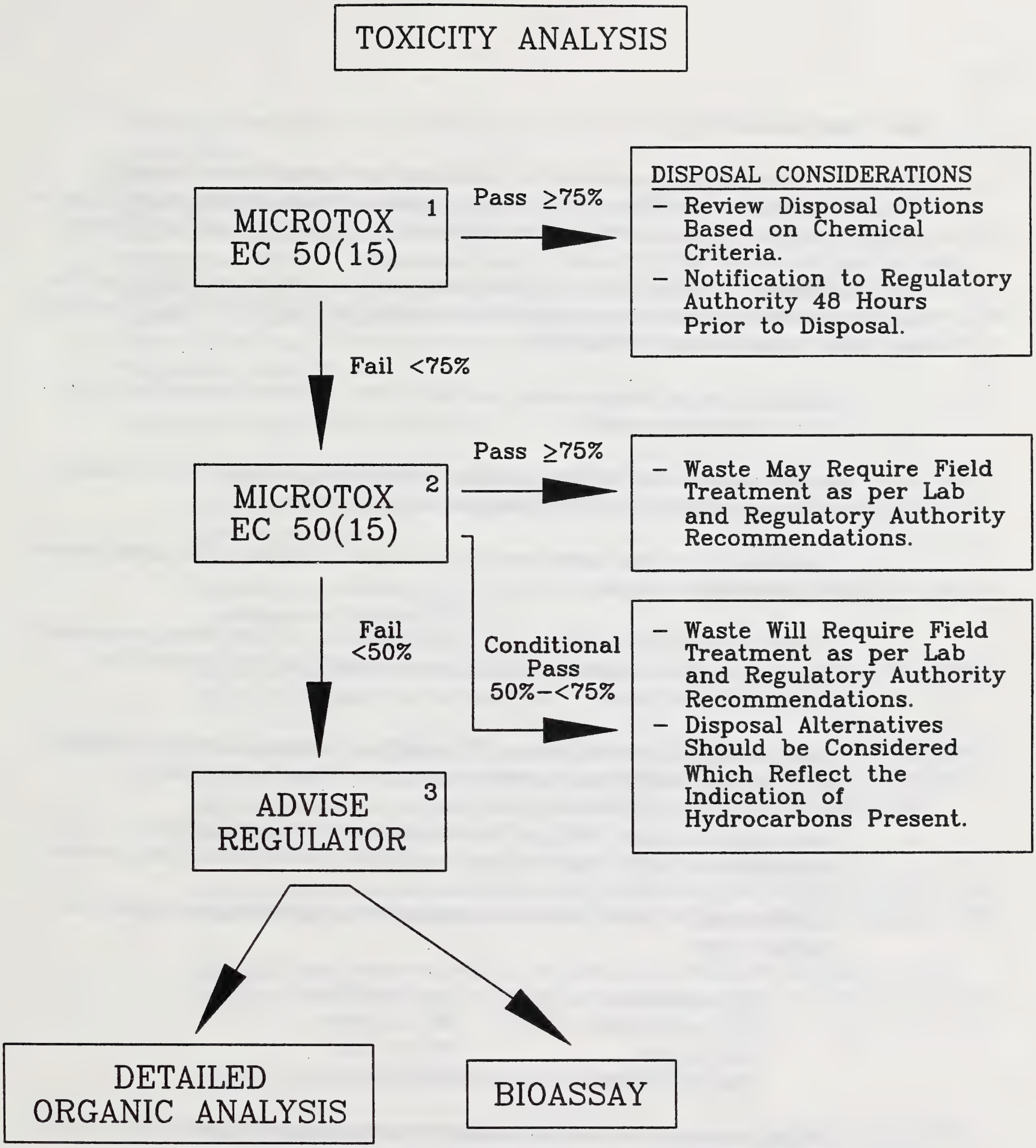
  Reporting units for the bioluminescent bacteria test are EC50(15) values.

## Disposal Criteria

*Mix / bury / cover*, and *landspreading* are the most common methods of drilling waste disposal. Therefore, specific criteria have been set for these two methods.

For privately owned land, an operator may proceed with waste disposal by these methods, two working days after receipt of the required waste characterization and disposal information by the appropriate ERCB field office. Landowner approval is required prior to disposal.

For publicly owned land, an operator may proceed with waste disposal by these methods, unless otherwise notified, five working days after receipt of the required waste characterization and disposal information by the appropriate public land management agency.


TOXICITY ANALYSIS
MICROTOX EC 50(15) 1
Pass ≥75%
DISPOSAL CONSIDERATIONS
– Review Disposal Options Based on Chemical Criteria.
– Notification to Regulatory Authority 48 Hours Prior to Disposal.
Fail <75%
MICROTOX EC 50(15) 2
Pass ≥75%
– Waste May Require Field Treatment as per Lab and Regulatory Authority Recommendations.
Fail <50%
Conditional Pass 50%–<75%
– Waste Will Require Field Treatment as per Lab and Regulatory Authority Recommendations.
– Disposal Alternatives Should be Considered Which Reflect the Indication of Hydrocarbons Present.
ADVISE REGULATOR 3
DETAILED ORGANIC ANALYSIS
BIOASSAY

1. Test Performed on Clarified Sample
2. Test Performed on Clarified Sample Treated with Activated Charcoal.
3. Discussion of Additional Testing and/or Disposal Options with Regulatory Authority is Required.

FIGURE 3: TOXICITY EVALUATION FOR DRILLING WASTE LIQUIDS
Refer To Appendix 1 For Procedures

### 1. Mix / Bury / Cover

This procedure is generally conducted after liquids have been pumped out and properly disposed. Liquids may be disposed with the solids providing that the mixing results in a stable material. Disposal is based on the analytical data for the combined liquid and solid slurry.

The goal of *mix / bury / cover* is to incorporate wastes meeting the required criteria into the soil at a level below the major rooting zone for plants but above the water table. Criteria for *mix / bury / cover* of wastes (Table 5) are:

- base of the final soil/waste mixture must be at least 1 m above the water table or a layer of permeable material,
- subsoil and waste must be mixed at least 1:1 (by volume) in order to ensure that the soil/waste mix is stable enough to support heavy equipment,
- a minimum 1 m of clean subsoil must be placed over the mixed solids; topsoil/surface soil must then be replaced,
- oil content must be less than 0.1% in the soil/waste mix; if these levels are exceeded a disposal plan will be required which addresses degradation of the hydrocarbon component of the waste,
- soil/waste mix pH must be between 6.5 and 8.5; on forest soils having an initial pH of 6.5 or lower, the pH increase of the soil/waste mix must not exceed 1.5,
- there must be less than 1600 kg of chloride disposed on the lease and the concentration in the soil/waste mix must not exceed 2,000 mg chloride/kg dry solids,
- where analysis for metals is required, metal content of the waste at a disposal site shall not exceed:

|  |  |  |  |
|---|---|---|---|
| 10 | kg boron | 3 | kg cadmium |
| 200 | kg chromium | 400 | kg copper |
| 200 | kg lead | 50 | kg nickel |
| 200 | kg vanadium | 600 | kg zinc, |

- if liquids are mixed with solids, nitrates plus ammonium (as N) must be less than 400 kg per site.

### 2. Landspreading

Operators are encouraged to contact the local reclamation officer and the landowner prior to disposal to ensure that the appropriate soil horizon is being used. The goal of *landspreading* is to dispose of the waste in a manner that preserves soil chemical, biological and physical properties by preventing the accumulation of salts, and to control the biological uptake and mobility of heavy metals. The landspread waste must not significantly affect the soil or groundwater.

The chemical and physical criteria for *landspreading* will be used when either liquids or solids are to be disposed on soil. Application of liquids must not result in pooling or runoff, and the total suspended solids content must not exceed 10,000 mg/L if the liquid is being applied to vegetation without any additional treatment. Where liquids and solids are to be disposed of on the same site, the combined loading rates must not exceed the criteria specified below. In any case, the spread rate for a waste must be based on the most limiting analyte in the waste or soil/waste mix.

Criteria for *landspreading* (Table 6) are:

- no more than 10 cm of waste solids should be applied in order to ensure complete mixing of the waste and the receiving soil, and to minimize potential effects on soil physical properties,
- the waste is to be thoroughly mixed into the soil at a minimum 1:3 waste/soil ratio to a maximum soil depth of 30 cm,
- pH of the soil/waste mix must be between 6.5 and 8.5; for forest soils with an initial pH of 6.5 or lower, the increase in pH of the soil/waste mix must not exceed 1.5,
- oil content in the soil/waste mix must be less than 0.5% in the upper 20 cm (topsoil), and less than 0.1% in the soil below 20 cm (subsoil); if these levels are exceeded a disposal plan will be required which addresses degradation of the hydrocarbon component of the waste,
- EC of the soil/waste mix must be less than 4 dS/m or, where original soil EC is more than 4 dS/m, the soil/waste mix can be up to 1 dS/m higher than the original soil EC,
- SAR of the soil/waste mix must be less than 10 or, where the original soil SAR is more than 10, the soil/waste mix must not exceed the original soil SAR by more than 1,
- chloride addition to the soil should not exceed 800 kg/ha,
- where metals analysis is required, metals addition to the soil should not exceed the following rates:

| | | | |
|---|---|---|---|
| 5 | kg/ha boron | 1.5 | kg/ha cadmium |
| 100 | kg/ha chromium | 200 | kg/ha copper |
| 100 | kg/ha lead | 25 | kg/ha nickel |
| 100 | kg/ha vanadium | 300 | kg/ha zinc. |

- if liquids are to be landspread, nitrate plus ammonium (as N) addition to the soil should not exceed 400 kg/ha.

## Special Cases

### Hydrocarbon Based Mud Systems

Landfarming of wastes with elevated oil content (hydrocarbon based systems) requires approval of a disposal and management plan submitted to the ERCB with the well license application. The ERCB will direct the plan to the appropriate regulatory agency if the well is located on public land.

Although the hydrocarbon component is generally considered to be bio-degradable, experience indicates that considerable care and management are required to promote the necessary biological activity under Alberta's soil and climatic conditions. Burial and subsurface spreading of this waste type is not an acceptable disposal practice, and operators are required to approach the disposal in a manner which will promote biodegradation of the hydrocarbon component.

When drilling conditions result in the unforeseen production of hydrocarbons, the plan must be submitted and approved prior to waste disposal.

Landfarming is required where the soil/waste mix is predicted to have an initial oil content over 0.5% in the top 20 cm of the topsoil or over 0.1% in the soil below 20 cm depth.

### High Salt Based Mud Systems

Disposal of wastes predicted to exceed the chloride levels in Tables 5 and 6, or nitrate (as N) levels given in Table 5 also require approval of a disposal and management plan submitted to the ERCB at the time of well licence application. The ERCB will direct the plan to the appropriate regulatory agency if the well is located on public land. When the final salt content of the waste is elevated due to penetrating a zone of high salt concentration during drilling, the disposal and management plan must be submitted and approved prior to waste disposal.

### Alternate Disposal Options

The option of subsurface disposal of drilling waste liquids in accordance with ERCB Interim Directive ID 81-1 has been retained, provided that the disposal zone is deeper than the deepest potable water zone. Operators considering this option should refer to the specific requirements in ERCB Interim Directive ID 81-1.

Other disposal options, such as the use of innovative disposal technology or process equipment must be approved by the appropriate regulatory agency, and in these cases a detailed plan must be provided that will meet environmental protection and reclamation requirements.

Operators are encouraged to review multi-well programs with the regulatory agency to avoid redundant sampling and analysis procedures. However, all or selected parts of these guidelines may be required by the approval agency.

## Reporting Requirements

The required waste characterization and disposal information will be summarized on a form published by the ERCB, and submisssion of the form will constitute a commitment by the licensee that disposal will meet or exceed the criteria of this Directive.

All data collected, supporting documents, and approvals must be held on file by the well owner until such time as a final reclamation certificate is obtained. Generally, there are two types of data to be retained:

- information forwarded to regulatory authorities should include; regulatory site disposition approval, drilling sump site condition reports, environmental assessments, sump liquid and solids analytical data, treatment summaries, reclamation certificates, and post-reclamation monitoring reports,

- records retained by the operator should include; sump design, construction summary, drilling mud summary, disposal methods summary, and post-reclamation summary.

Table 1 - Liquid Phase Analytical Methods

## 1.0 SAMPLE PREPARATION

Pass the sample through a 0.45 µm filter and use for the following analyses (except for 2.9 where the whole sample is used, and 2.10 where centrifuging is required).

## 2.0 CHARACTERIZATION

| Analytical parameter and reporting units | Procedure | Reference |
|---|---|---|
| 2.1 pH | Combination electrode and pH meter. | Method 4500 h in APHA (1992). |
| 2.2 Specific conductance (dS/m) | Conductivity cell and conductance meter. | Method 2510 in APHA (1992). |
| 2.3 Ca, Mg, Na, K (mg/l) | All ions can be measured by Inductively Coupled Atomic Emission Spectroscopy (ICP-AES) or Atomic Absorbtion (AA). Flame Photometry can be used for Na and K only. | Methods 3500 C for ICP-AES, 3500 B for AA, or 3500D for flame photometry in APHA (1992). |
| 2.4 Chloride (mg/l) | Colorimetric measurement using Flow Injection (FIA), Autoanalyzer®, or Ion Chromatography (IC). | Methods 4500-CL E (colorimetric) or 4500-CL F (IC) in APHA (1992). |
| 2.5 $N0_3$ (mg/l) | Colorimetric measurement using Flow Injection (FIA), Autoanalyzer®, or Ion Chromatography (IC). | Methods 4500-$N0_3$ E or F in APHA (1992). |
| 2.6 $SO_4$ (mg/l) | Colorimetric measurement using Flow Injection (FIA), or Ion Chromatography (IC). | Method 4500-$SO_4$ F (colorimetric) or 4500-$SO_4$ B in APHA (1992) |

| | | | |
|---|---|---|---|
| 2.7 | $NH_4$ (mg/l) | Manual colorimetric measurement using Flow Injection (FIA), Autoanalyzer®, or Ion Chromatography (IC). | Method 4500-$NH_4$ B followed by 4500-$NH_4$ D, or 4500-$NH_4$ H in APHA (1992). |
| 2.8 | Sodium Adsorption Ratio (SAR) | Calculated from soluble ions (Ca, Mg, and Na in 2.3). | Method 3.26 in McKeague 1978. |
| 2.9 | Total Suspended Solids (TSS) (mg/l) | Filter through a porcelain crucible containing preignited glass fibre filter (1.2 µm). The residue retained by the glass fibre filter is dried to a constant weight at 105 °C for one hour. | Method 2540 D in APHA (1992). |
| 2.10 | Microtox® bioassay | Measure EC20 and EC50 at both 5 and 15 minutes. | ERCB (1992), appended in Appendix 1. |

Table 2 - Solid Phase Analytical Methods

## 1.0 SAMPLE PREPARATION

Air dry (less than or equal to 2% water content) and grind. Collect a representative portion passing a 2 mm stainless steel sieve.

## 2.0 CHARACTERIZATION

| Analytical parameter and reporting units | | Procedure | Reference |
|---|---|---|---|
| 2.1 | Water content ($\Theta_m$) (grams water ÷ grams dry solids) | Oven dry | Method 2.411 in McKeague (1978) |
| 2.2 | pH (I)<br>pH (II) | Water paste (2.3 extract)<br>2:1 slurry of 0.01 M $CaCl_2$. Use combination electrode and pH meter. | Methods 3.14 and 3.11 in McKeague (1978). |
| 2.3(a) | Saturated paste and paste solution | Saturated extract is prepared by USDA Soil Salinity lab method, filtered through 0.45 μm filter and the extracts used for analyses 2.4 to 2.7. | Method 3.21 in McKeague (1978). |
| 2.3(b) | Percent saturation (%) | Oven dry a soil paste sample. | Method 3.21 in McKeague (1978). |
| 2.4 | Electrical conductivity of 2.3 extract (dS/m) | Same as 2.2 in Table 1. | Same as 2.2 in Table 1. |
| 2.5 | Chloride in 2.3 extract (mg/l) | Same as 2.4 in Table 1. | Same as 2.4 in Table 1. |
| 2.6 | Ca, Mg, Na, K in 2.3 extract (mg/l) | Same as 2.3 in Table 1. | Same as 2.3 in Table 1. |
| 2.7 | SAR of 2.3 extract | Calculated as in 2.8 in Table 1. | Same as 2.8 in Table 1. |
| 2.8 | Hot water soluble Boron (mg/kg) | ICP-AES or curcumin colour | Methods 4500-B D or 4500-B B in APHA (1992) |

| | | | |
|---|---|---|---|
| 2.9 | Corrected wet bulk density ($g/cm^3$) | Calculated from the (weight of dry solids) ÷ (volume of wet waste).<br>or<br>Estimated from the water content ($\Theta_m$) and density of solids as follows:<br>$\frac{1}{\Theta_m + 0.38}$<br>(This assumes that oil content of waste is negligible and that water content is at or above soil paste condition.) | Abboud, 1993 |
| 2.10 | Metals analysis (Cr, V, Cd, Cu, Pb, Zn, Ni) (mg/kg) | Hot acid ($HN0_3$-HF) digestion in closed teflon vessels under microwave heating followed by ICP-AES or AA measurements. | Digestion as in Abboud (1991) and Appendix 1, and metals using methods 3500 C (ICP) or B (AA) in APHA (1992). |
| 2.11 | Oil content (%) | Measured gravimetrically by Soxhlet Extraction with methylene chloride*. | McGill & Rowell 1977. |
| 2.12 | Texture | Hand method | Method 4.8 in McKeague (1978) |

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* The determination of oil content using this method may provide false positive values, due to the presence of substances such as tannins and humic or fulvic acids. Corrections for these substances should be made (Refer to Method 5510-C in APHA (1992)).

Table 3 - Soil Analytical Methods

## 1.0 SAMPLE PREPARATION

Air dry (less than or equal to 2% water content) and grind. Collect a representative portion passing a 2 mm stainless steel sieve.

## 2.0 CHARACTERIZATION

| | Analytical parameter and reporting units. | Procedure | Reference |
|---|---|---|---|
| 2.1 | pH (I)<br>pH (II) | Water paste (2.2 extract)<br>2:1 slurry of 0.01 M $CaCl_2$. Use combination electrode and pH meter. | Methods 3.14 and 3.11 in McKeague (1978). |
| 2.2(a) | Saturated paste and paste extract solution | Saturated extract is prepared by USDA Soil Salinity lab method, filtered through 0.45 µm filter and the extracts used for analyses 2.1, 2.3 to 2.6. | Method 3.21 in McKeague (1978). |
| 2.2(b) | Percent saturation (%) | Oven dry a soil paste sample. | Method 3.21 in McKeague (1978). |
| 2.3 | Electrical conductivity of 2.2 extract (dS/m) | Same as 2.2 in Table 1. | Same as 2.2 in Table 1. |
| 2.4 | Ca, Mg, Na, K in 2.2 extract (mg/l) | Same as 2.3 in Table 1. | Same as 2.3 in Table 1. |
| 2.5 | SAR of 2.2 extract | Calculated as in 2.8 in Table 1. | Same as 2.8 in Table 1. |
| 2.6 | Chlorides in 2.2 extract (mg/l) | Same as 2.4 in Table 1. | Same as 2.4 in Table 1. |

**Table 4.** Levels of Metal Addition (in kilograms) to Drilling Muds That Will Require Metals Analysis of Waste Solids.

| | |
|---|---|
| Boron | 2.5 |
| Cadmium | 0.75 |
| Chromium | 50 |
| Copper | 100 |
| Lead | 50 |
| Nickel | 12.5 |
| Vanadium | 50 |
| Zinc | 5 |

Table 5. Criteria for *Mix / Bury / Cover* of Drilling Waste Solids

| **Parameter** | **Criteria for Waste/Soil Mixture** |
|---|---|
| pH | 6.5 to 8.5, or no more than a 1.5 increase on forested soils with an initial pH of less than 6.5 |
| Oil content | < 0.1 % |
| Chloride | < 1600 kg per site and < 2000 mg Cl/kg dry solids |
| Metals* | Boron 10 kg<br>Cadmium 3 kg<br>Chromium 200 kg<br>Copper 400 kg<br>Lead 200 kg<br>Nickel 50 kg<br>Vanadium 200 kg<br>Zinc 600 kg |
| Nitrate plus ammonium (as N) | 400 kg per site (if liquids buried) |

* Values listed denote levels before waste/soil mixing.

Table 6. Criteria for *Landspreading* Drilling Wastes.

| **Parameter** | **Criteria for Waste/Soil Mixture** |
|---|---|
| Electrical Conductivity (EC) | 4 dS/m or, where original soil EC is more than 4 dS/m, the soil/waste mix can be up to 1 dS/m higher than the original soil EC. |
| pH | 6.5 to 8.5, or no more than a 1.5 increase on forested soils with an initial pH of less than 6.5 |
| Oil content | < 0.5 % in upper 20 cm of soil and < 0.1 % below 20 cm. |
| Chloride | 800 kg/ha |
| Metals* | Boron 5 kg/ha<br>Cadmium 1.5 kg/ha<br>Chromium 100 kg/ha<br>Copper 200 kg/ha<br>Lead 100 kg/ha<br>Nickel 25 kg/ha<br>Vanadium 100 kg/ha<br>Zinc 300 kg/ha |
| Sodium Adsorption Ratio (SAR) | 10 or background. If background is higher than 10, the waste/soil mix must not exceed the original soil SAR by more than 1. |
| Nitrate plus ammonium (as N) | 400 kg/ha |

* Values listed denote levels before waste/soil mixing.

## Glossary

| | |
|---|---|
| Composite Sample | A sample consisting of a number of thoroughly mixed samples in which each contributes equally to the composite. Composite samples provide an estimate of the mean of the population from which the samples forming the composite are drawn. |
| Equivalent Land Capability | The ability of the land to support various land uses after reclamation, must be similar to the ability that existed prior to an activity being conducted on the land, although the ability to support individual land uses will not necessarily be identical after reclamation. |
| Landfarming | A waste treatment/disposal method whereby single or multiple applications of biodegradable wastes are made on a dedicated parcel of land which is managed in a manner which allows the soil system to degrade, transform and assimilate the waste constituents. |
| Landspreading | A disposal method whereby the waste is spread over a predetermined land area based on an acceptable loading rate. Landspreading is usually done for wastes containing elevated levels of heavy metals and/or salts, and involves a single application. The wastes are normally incorporated into the soil. |
| Liquids | Components of drilling sump contents that contain less than 5% total suspended solids (TSS). |
| Mix / Bury / Cover | A method whereby sump solids are stabilized and diluted by mixing with subsoil. The waste materials must be mixed at least 1:1 by volume with the subsoil. The stable waste/soil mixture is then placed into the original sump, or other excavations, and is covered with at least one meter of clean subsoil, and then the original topsoil. |
| Solids | Components of drilling sump contents that contain equal to or greater that 5% total suspended solids (TSS). |

## References

1. Abboud, S.A., 1991. Total heavy metal analysis methods: Comparison of various techniques. Proceedings of the 4th annual WEALA workshop: Heavy Metals in the Environment. Edmonton.

2. APHA (American Public Health Association), 1992. Standard Methods for the Examination of Water and Wastewater. 18th Ed. Washington.

3. ERCB, 1992. ERCB Microtox® bioassay method. ERCB, Calgary, Alberta.

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5. McKeague, J.A. (Ed.), 1978. Manual on soil sampling and methods of analysis. 2nd Edition. Prepared by the Subcommittee on Methods of Analysis of the Canada Soil Survey Committee, Canadian Society of Soil Science. Ottawa. 212 pp.

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# APPENDIX 1: MICROTOX® BIOASSAY PROCEDURE

## 1.0 SUMMARY

Toxicity is the tendency of a chemical or condition to cause harm to the life process. The minimum requirement for monitoring toxicity is a set of inter-dependent enzyme systems controlling measurable physiological parameters, along with the appropriate measurement system. For a practical, real-time measurement of acute toxicity, a biological system must be selected which can provide a simple, sensitive and rapid measurement.

The Microbics Microtox® system fulfils specific conditions for acute toxicity assessment for a spectrum of chemical compounds; it is intended for use in determining acute toxicity in aqueous Microtox® reagent, which is living bio-luminescent bacteria that have been harvested under strict growing conditions and then lyophilized (freeze dried under vacuum). At the time of testing, the lyophilized bacteria are reconstituted to provide a ready to use cell suspension. The production of light by the organisms is a reflection of their state of health. The Microtox® System directly measures the inhibition of life sustaining physiological processes caused by toxic substances.

The Microtox® analyzer, which is simply a precision photometer coupled with a small refrigerator and recorder, is used to make quantitative measurements of the response of the living bio-luminescent bacteria to toxic samples, as evidenced by changes in the light produced by the bacteria.

## 2.0 APPLICATION

Drilling waste liquids usually contain complex mixtures of both organic and inorganic constituents used in conventional drilling operations. Toxicity bioassays of these wastes are used to determine if components toxic to vegetation, micro-organisms, aquatic species as well as animals or humans might be present, but are not detected in routine chemical analysis. The bioassay is a screen or early warning system which will alert the analyst, field operator and regulator that certain toxic constituents may be present. The bioassays which are suited for this application must be responsive to small changes in concentrations of components which are organic, metallic, organo-metallic or gaseous, and should not be dependant on nutrient or major ion concentrations.

Bioassay results can be of assistance in developing and evaluating waste treatment and disposal options.

The main purposes of conducting bioassays on drilling wastes are:

- To determine the presence or absence of toxic constituents.

- To ascertain the nature, extent and probable causes of toxicity.

- To assist in developing and evaluating waste treatment options, and in making disposal decisions.

The methodology described herein is applicable to biological toxicity assessment of sump fluids, domestic water wells, chemical additives, soil and vegetation extracts.

## 3.0 SAMPLE HANDLING AND PRESERVATION

3.1 Samples should be field collected in clean glass containers. Plastic containers are unsuitable due to the presence of toxic antioxidants, plasticizers and/or mould releasing agents. Some plastics could absorb certain toxicants.

3.2 Samples which will not be tested immediately should be refrigerated at 4°C. Analysis should be carried out within 72 hours.

3.3 Sample Clarification

3.3.1 The sample must be representative of the drilling waste being considered for disposal. Most samples will consist of liquid and solid phases and may range from clear to slightly turbid water or moderate to highly turbid thick slurry.

3.3.2 The Microtox® bioassay must be performed on a clarified portion of the sample as received. Samples with a turbidity $>10$ NTU must be clarified prior to analysis. Clarification can usually be achieved through settling and decantation, or by centrifuging. Choose a centrifuge speed and time period which is sufficient to clarify the test sample to $<10$ NTU. A speed of 5000 - 15,000 rpm may be required.

3.3.3 Filtration of sump fluids for the purpose of clarification should be avoided, as toxic constituents may be removed from the sample through adsorption on the filtering membrane.

3.3.4 Visible hydrocarbon layers or sheens must be removed from the sample prior to analysis. This can be accomplished by use of a separatory funnel.

3.4 Measure the pH, and if outside the range 6.0 - 8.8, alter using 0.1N $H_2SO_4$ or 0.1N NaOH. Stronger solutions of acid/base may be used on highly buffered samples. Adjust pH to as near 7.0 as possible (6.8 - 7.2).

3.5 Store Diluent, Reconstituting Solution and Osmotic Adjusting Solution (MOAS) in the refrigerator at 4°C. The reagent (luminescent bacteria) should be stored in a freezer until just prior to using. It is recommended that a non-frost free freezer be used.

## 4.0 APPARATUS AND MATERIALS

4.1 Microbics Model 500 or 2055 Microtox® Analyzer or equivalent.

4.2 Strip - Chart Recorder.

4.3 Micropipettors: 10 µl, 500 µl, 200 µl, 1000 µl with disposable plastic tips.

4.4 Microtox® Reagent (vial of freeze-dried bacteria).

4.5 Microtox® Diluent (2% NaCl solution).

4.6 Microtox® Reconstituting Solution (sterile demineralized water).

4.7 Microtox® Osmotic Adjusting Solution (MOAS): 22% NaCl solution.

4.8 Microtox® Standard Cuvettes (11.75 mm x 50 mm).

4.9 Sharp Model 5100 Scientific Calculator or computer software program.

## 5.0 INTERFERENCES

5.1 The following conditions are known to give either a positive (toxic) or a negative (less toxic) response to the Microtox® bioassay.

5.1.1 Highly turbid samples - clarify prior to analysis, section 3.3.

5.1.2 Highly coloured samples - colour correction techniques as described by the instrument manufacturer should be followed. (Reference 11.1)

5.1.3 Hydrocarbon film - remove by sample clarification techniques, section 3.3.4.

5.1.4 Decomposition products found in stagnant putrefying water.

5.1.5 Biodegradation of the sample over time and temperature.

5.1.6 Adsorption of toxicants onto particulates, or onto the filtering medium during sample filtration.

5.2 Samples rich in nutrients that are compatible with the luminescent bacteria can cause an increase in light output during the testing period of 15 minutes.

# 6.0 PROCEDURE

## 6.1 ANALYZER PREPARATION

The following steps are necessary to ensure that the analyzer and recorder are operational. Ensure that partially filled cuvettes are left in the pre-cooling well and incubator wells at all times. Please note that these instructions apply only to the Microbics Model 2055. Consult the manual for appropriate instructions for the Model 500.

6.1.1 Check the temperature readings of the analyzer by pressing the appropriate controls on the instrument panel.

| Control | Temperature Reading |
|---|---|
| TURRET | 15° ± 0.2°C |
| INCUB | 15° ± 0.2°C |
| AIR | 3.0° ± 1.0°C |

6.1.2 Press HV CHECK; voltage reading should fall between -500 to -900.

6.1.3 With HV, RUN and XI depressed, set SPAN (100% ADJ) dial to 999. Turret should be open.

6.1.4 Adjust ZERO (ZERO% ADJ) control until the minus sign on the DIGITAL PANEL METER (DPM) flashes. Lock control.

6.1.5 Adjust SPAN (100% ADJ) to 400, lock. Press X10 SENSITIVITY, close turret. Press CAL CHECK, ensure DPM reading falls between 90 - 110. Reset SENSITIVITY to XI.

6.1.6 If the above readings fall outside the specification range, consult the instrument manual for remedial action.

6.1.7 Recorder adjustments

6.1.7.1 With turret open, adjust RECORDER ZERO control so that the pen traces on zero on the chart paper.

6.1.7.2 Ensure in step 6.1.5 that the recorder pen traces to >90 on the hart paper.

## 6.2 PROCEDURE

| | | | | | |
|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 |
| PRE-COOLING WELL | 6 | 7 | 8 | 9 | 10 |
| | 11 | 12 | 13 | 14 | 15 |
| | | INCUBATOR WELLS | | | |
| TURRET | | | | | |

6.2.1 Fill the pre-cooling and incubator wells with new, clean cuvettes.

6.2.2 Bring out Diluent, Reconstituting solution and MOAS from the fridge. Take the reagent from the freezer and store in the fridge (4°C) about 15 minutes before using.

6.2.3 Pipet 1.0 ml (1000 µl) of Microtox® Reconstituting Solution into the pre-cooling well cuvette.

6.2.4 Pipet the following volumes into the incubator well cuvettes.

6.2.4.1 Pipet 2000 µl sample and 200 µl of MOAS into cuvette 5, mix by aspirating and dispensing the liquid approximately 5 times.
NOTE: The sample concentration has now been diluted to 90.9%. This dilution factor must be considered when the final sample concentration is calculated. (Sections 6.2.6 and 6.2.18).

6.2.4.2 Pipet 1000 µl diluent into cuvettes 1 to 4 and cuvette 6.

6.2.4.3 Pipet 500 µl diluent into cuvettes 7 to 15.

6.2.5 Prepare 1:1 serial dilutions of the sample as follows

6.2.5.1 Pipet 1000 µl from well 5 into 4, mix by aspirating and dispensing the liquid approximately 5 times.

6.2.5.2 Pipet 1000 µl from well 4 into 3, mix.

6.2.5.3 Pipet 1000 µl from well 3 into 2, mix.

6.2.5.4 Pipet 1000 µl from well 2 into 1, mix.

6.2.5.5 Discard 1000 µl from well 1.

6.2.6 The sample assay concentrations at this point are

| Cuvette No. | Concentration |
|---|---|
| 1 | 5.68% |
| 2 | 11.36% |
| 3 | 22.73% |
| 4 | 45.46% |
| 5 | 90.91% |

6.2.7. To assure temperature equilibration, wait 5 minutes.

6.2.8 Take one vial of Microtox® Reagent from the refrigerator and discard the rubber stopper and seal. When opening, make sure the vial was under vacuum, and when open, that the pellet is flat on the bottom of the vial. Pour the reconstitution solution from the pre-cooling well cuvette rapidly into the vial. Mix the vial for a few seconds while holding the vial at the top, to minimize warm-up. Pour the reconstituted reagent back into the same cuvette and immediately return the cuvette to the pre-cooling well. Mix the reconstituted reagent with a 500 µl micropipettor by aspirating and dispensing 500 µl of the solution about 20 times. Keep the cuvette in the pre-cooling well during this mixing step.

6.2.9 Using a micropipettor, add 10 µl of reconstituted reagent from the pre-cooling well into cuvettes 7 to 15. Mix each cuvette 5 times using a 500 µl micropipette.

6.2.10 To assure reagent equilibration, wait 15 minutes. This can be done by using the recorder set at a chart speed of 1 cm/min. and measuring the distance required for the 15 minute time period. A stopwatch may also be used.

6.2.11 Prior to the elapsed 15 minute waiting period (or at about the 7-10 minute mark), place cuvette 7 in the turret, turn on recorder, and close the turret. Adjust the recorder pen to read approximately 70-80% on the chart paper by adjusting the SPAN (100% ADJ) control. The ANALYZER and RECORDER PREPARATION STEPS as described in section 6.1 should be carried out immediately prior to this step.

6.2.12 Record (trace) the light level from cuvette 7 for about 2 seconds. Remove cuvette from the turret and return to incubator well.

6.2.13 Proceed to cycle cuvettes 8-15 as described above, recording the light level for each cuvette for about 2 seconds.

6.2.14 When complete, choose the 6 cuvettes with the closest light readings and place them in incubator wells 10-15. Place the 3 extra cuvettes in wells 7, 8, 9. These 3 cuvettes will not be used in the remainder of the analysis.

6.2.15 At this time, note the dial setting on the SPAN (100% ADJ) control. This reading will give the operator an indication as to the health and stability of the reconstituted reagent. A reading of 200 - 500 indicates healthy and relatively stable bacteria, whereas readings of greater than 700 seem to indicate unstable or unhealthy bacteria. Repeat steps 6.2.8 to 6.2.14 using fresh Microtox® Reagent if the readings exceed 700.

6.2.16 When the 15 minute time elapses (Step 6.2.10), cycle cuvettes 10 - 15 as follows: transfer cuvette 10 into the turret, close handle and adjust recorder tracing to equal 85% by use of SPAN (100% ADJ) control, then lock dial. Continue to record cuvettes 11 - 15. This is the initial light level (I-0) for the test.

6.2.17 Immediately pipet 500 µl of reagent blank (from cuvette 6) and also from test samples (cuvettes 1-5) into cuvettes 10 - 15, described as follows:

| | | | | | |
|---|---|---|---|---|---|
| Pipet | 500 µl | from | 6 | into | 10 |
| " | " | " | 1 | " | 11 |
| " | " | " | 2 | " | 12 |
| " | " | " | 3 | " | 13 |
| " | " | " | 4 | " | 14 |
| " | " | " | 5 | " | 15 |

Use the same pipet tip, and mix each transfer by aspirating and dispensing 500 µl volumes 5 times. Do quickly. Keep all cuvettes in the incubator wells during mixing.

6.2.18 The final sample concentrations are:

| Cuvette | Concentration |
|---|---|
| 11 | 2.8% |
| 12 | 5.6% |
| 13 | 11.2% |
| 14 | 22.5% |
| 15 | 45.0% |

6.2.19 Exactly five and fifteen minutes after the addition of reagent blank into cuvette 10 (Step 6.2.17), cycle cuvettes 10 - 15 into the turret, recording light levels for about 2 seconds each. These readings represent 5 minute (I-5) light levels and fifteen minute (I-15) light levels for the test sample and blank. The recorder or a stopwatch can be used to time the 5 and 15 minute readings.

6.2.20 Record I-0, I-5, and I-15 light levels obtained for the reagent blank and each test sample dilution, and transfer readings onto the data assay sheet. Readings are taken directly from the chart paper, expressed as peak height measurements, and are reported to the nearest 0.5 unit.

6.2.21 Use the programmed calculator Sharp Model 5100 to calculate the gamma ($\Gamma$), or light loss values, for I-0, I-5 and I-15.

6.2.22 Using log paper, graph the $\Gamma$ values for each sample dilution, and calculate the EC 20 and EC 50 concentrations for the 5 and 15 minute time frames. Detailed instructions for calculating EC 20 and EC 50 values are included in section 9.

6.2.23 Alternative EC 20 and EC 50 calculations can be made on a variety of computer software calculator programs. Please contact the Energy Resources Conservation Board (ERCB) Chemical Research Laboratory for further information, or refer to instructions provided by the manufacturer.

## 7.0 HEALTH AND SAFETY

7.1 Standard safe laboratory operating procedures should be followed at all times when performing the Microtox® bioassay. Test samples may contain hazardous or toxic substances.

7.2 Equipment operators should wear appropriate safety clothing (lab coat, safety glasses, and gloves). Safe hygienic practices should be followed. Fume hoods should be used when required.

7.3 Hazardous test samples should be disposed of in a safe and approved manner.

7.4 The Microtox® Reagent (lyophilized bacteria) has not been found to be pathogenic to humans.

## 8.0 QUALITY CONTROL: PRECISION AND ACCURACY OF RESULTS

8.1 Each analytical run should include approximately 10% of all effort on Quality Control to ensure confidence in the data. The Quality Control procedure on each analytical run normally includes: replicate determinations on selected samples to measure precision, and analysis of a reference standard material to check the accuracy of the run and the sensitivity of the luminescent bacteria. Two preferred reference standard materials are solutions of phenol and zinc sulphate. Their preparation is described as follows:

8.1.1 Phenol Standard Solution - dissolve 0.1316 gm. of ACS reagent grade phenol crystals in high purity demineralized water (equivalent to Millipore Milli-Q water), and make up to 1 litre in volume. Store this solution at 4°C.

8.1.2 Zinc Sulphate Standard Solution - dissolve 0.0881 gm. of ACS reagent grade $ZnSO_4 \bullet 7H_2O$ in high purity demineralized water (equivalent to Millipore Milli-Q water), and make up to 1 litre in volume. Store at 4°C.

8.2 Participation in a regular Round Robin Quality Control program, such as provided by the Western Canada Microtox® Users Committee (WCMUC) is recommended.

## 9.0 CALCULATIONS

9.1 Results obtained using this method should be reported for EC 20 and EC 50 values for both the 5 and 15 minute time intervals.

The EC 20 (EC 50) value is defined as the concentration of the original test sample which causes a 20% (or 50%) loss of light from the test bacteria.

The units of concentration applicable to the EC 20 and EC 50 values are usually expressed in % concentration of the original test sample, as it applies to sump fluids, drilling wastes, soil and vegetation extracts, etc. However, some test samples may be reported as mg/L (ppm) if so desired. This would apply to reference standard material, such as phenol or zinc solutions described earlier.

EC 20 and EC 50 values obtained from graphical, calculator or computer calculations which have been extrapolated past 100% concentration should be expressed as >100%.
ie: EC 50 (15) = >100%
This represents a non-toxic response.

Similarly, the light output readings on the recorder will show very little or no light loss for the I-0, I-5, and I-15 readings for certain samples. The Γ (gamma) calculations for these readings will typically range from 0.01 - 0.10 over the test sample concentration range. The EC 20 and EC 50 values in this case can be recorded as "no significant effect", which again represents a non-toxic response.

9.2 Gamma (Γ) calculations

Gamma (Γ) is the preferred measure of light loss. The Γ effect is the ratio of the light lost during test time (t) to the light remaining at the time (t).

### 9.2.1 Blank Response Calculations (Blank Ratio)

The Microtox® procedure requires that a non-toxic reagent blank be assayed concurrently with the test samples. This allows for minor changes in light output from the bacteria when they are exposed to non-toxic samples (for the time interval).

$$R(t) = \frac{I(t)\,b}{I\text{-}0\,b}$$

$R(t)$ = blank ratio for time (t)

$I\text{-}0\,b$ = initial light reading for the blank cuvette at 0 time.

$I(t)b$ = final light reading for the corresponding blank cuvette at (t) minutes.

### 9.2.2 Gamma (Γ) Effect Calculation

$$\Gamma(t) = \frac{R(t)\,I\text{-}0}{I(t)} - 1$$

$\Gamma(t)$ = the Γ effect calculated for exposure time (t).

$I\text{-}0$ = the initial light reading for any given test cuvette at "zero" time, just before the organisms are added to test sample.

$I(t)$ = The final light reading for the corresponding test cuvette at time (t).

Sample Calculations

Readings = Blank I-0 = 85

Blank I-5 = 81

Sample I-0 = 81.5

Sample I-5 = 54.5

$$R(t) = \frac{81}{85} = 0.95$$

$$\Gamma(t) = \frac{0.95(81.5)}{54.5} - 1$$

$$= 0.42$$

Gamma calculations are routinely carried out on the programmable calculator, Sharp Model 5100, or a variety of computer software programs.

### 9.2.3 Calculation of EC 20 and EC 50 Values

EC 20, (50) is defined as the effective concentration of toxicant (or test samples) causing a 20% (or 50%) decrease in the Microtox® Reagent light output under the defined condition of exposure time (t).

Normally EC 20 and EC 50 values are calculated graphically on log paper. The x-axis is plotted as sample concentration, while the y-axis is plotted as $\Gamma$ (t). A best fit line is drawn through the plotted points for the 5 and 15 minute $\Gamma$ values.

The EC 20 value corresponds to the sample concentration giving a $\Gamma$ value of 0.250.

The EC 50 value corresponds to the sample concentration giving a $\Gamma$ value of 1.0.

The EC 20 and EC 50 values can be calculated on the Sharp Model 5100 programmable calculator, or a variety of computer software programs.

## 10.0 DATA INTERPRETATION

10.1 ERCB staff have arrived at the following Microtox® data conclusions as a result of years of experience in comparing the EC 20 and EC 50 values to the standard 96 hour Rainbow Trout bioassay results.

It should be stated that Microtox® values cannot alone determine the toxicity of a sample, but must be considered only in addition to other factors such as sample pH, chloride and sulphate concentrations.

When comparing toxicity readings from two or more bioassay techniques, one must be aware that certain toxicants may affect one bioassay quite differently than the other.

ie: hydrocarbons are very toxic to the Microtox® bioassay, but could have very little effect on other bioassays such as the Rainbow Trout bioassay.

## 10.2 MICROTOX VALUES

| EC 20 | EC 50 | TOXICITY | TROUT BIOASSAY |
|---|---|---|---|
| 0-30 | 0-50 | Toxic | Dead Fish |
| 30-50 | 50-90 | Slight toxicity | Some fish dead at 'x' hours, loss of orientation |
| >50 | >90 | No significant effect - passes | Fish okay @ 96 hours |

Any sample pH adjustments and sample clarification techniques (decantation, centrifuging, removal of hydrocarbons) prior to analysis must be indicated when the analysis results are reported. Any laboratory treatment options as described in the attached addendum (charcoal, aeration, filtration) which were carried out must also be reported.

## 11.0 REFERENCES

11.1 Microtox® Manual, A Toxicity Testing Handbook, Microbics Corp.

11.2 Rapid Toxicity Test Using Luminescent Bacteria, Environment Protection, C & P, Environment Canada.

11.3 Strosher, M.T., A Comparison of Biological Testing Methods in Association with Chemical Analyses to Evaluate Toxicity of Waste Drilling Fluids in Alberta. Canadian Petroleum Association.

11.4 Standard Procedure for MICROTOX® Analysis, Western Canada Microtox® Users Committee (WCMUC), December 4, 1991.

11.5 Microtox® System Operating Manual, Beckman Instruments.

# ADDENDUM: MICROTOX® BIOASSAY METHOD
# LABORATORY TREATMENT OPTIONS

## INTRODUCTION

All sump fluids being considered for disposal are required to have a Microtox® bioassay performed to determine a toxic response, as outlined in the Interim Directive ID 93-1 Drilling Waste Disposal Guidelines.

For sump fluid samples which demonstrate a toxic response during the initial screening, appropriate laboratory treatment and re-testing is to be performed to provide additional information regarding the nature and probable cause of the observed toxicity.

Laboratory treatment options include the following:

1.0 Charcoal Treatment - will assist in determining whether the toxic components in the sample will adsorb in similar environmental conditions following disposal.

2.0 Sample Aeration - will indicate that the toxic components can be oxidized, volatilized or degassed.

3.0 Sample Filtration - will indicate that the toxic components can be absorbed onto filtering medium.

## PROCEDURE

### 1.0 CHARCOAL TREATMENT

#### 1.1 PREPARATION OF CHARCOAL

1.1.1 Use a good quality granular activated charcoal, such as bone or hardwood derived charcoal suitable for use in fish aquariums, 10-20 mesh size.

1.1.2 Prewash the charcoal by placing about 2 kg in a plastic colander or fine nylon mesh sieve and rinsing for several minutes with hot tap water followed by high purity demineralized water. Ensure all charcoal fines (dust) are removed during this step. Repeat rinsing step if necessary.

1.1.3 Dry the washed charcoal at 105 degrees Celsius in a clean, organic vapour free, drying oven for 16 hours.

1.1.4 Cool to room temperature and store in a clean, tightly closed glass container.

#### 1.2 TREATMENT PROCEDURE

1.2.1 Transfer 5 ml of a clarified, pH adjusted sample into a clean 10 ml glass test tube fitted with a polyethylene cap.

1.2.2 Add approximately 0.6 g of prepared activated charcoal.

1.2.3 Shake tube gently for about 10 seconds every 2 minutes for a total of 10 minutes.

1.2.4 Allow charcoal to settle for about 30 minutes. Perform the Microtox® bioassay on the clear supernate. Follow the ERCB Microtox® Bioassay Method as described in Interim Directive ID 93-1 Drilling Waste Disposal Guidelines.

### 1.3 QUALITY CONTROL

1.3.1 Test the fresh activated charcoal for the presence of toxicants on a regular basis by substituting high purity, toxicant free demineralized water for the test sample, and follow the procedure described in 2(a) to 2(d).

1.3.2 This procedure should be followed whenever

1.3.2.1 a new batch of activated charcoal is prepared.

1.3.2.2 a series of sump samples are tested.

1.3.3 Carry out the bioassay on the untreated demineralized water. This will serve as the analytical "blank".

## 2.0 SAMPLE AERATION

2.1 Aeration should be performed using ultra high purity (99.995%) oxygen obtained from a reliable supplier.

### 2.2 TREATMENT PROCEDURE

2.2.1 Transfer 10 ml of a clarified, pH adjusted sample into a clean 25 ml glass test tube.

2.2.2 Aerate with oxygen for about 5 minutes, at a rate of about 5 ml per minute, using care to prevent loss of sample.

2.2.3 Perform the Microtox® bioassay on the sample, following the ERCB Microtox® Bioassay Method as described in Interim Directive ID 93-1 Drilling Waste Disposal Guidelines.

### 2.3 QUALITY CONTROL

2.3.1 Test for the presence of toxicants from the oxygen source by aerating a 10 ml sample of high purity, toxicant free demineralized water.

2.3.2 Perform the Microtox® bioassay on the aerated demineralized water.

2.3.3 This procedure should be followed whenever

2.3.3.1 a new cylinder of oxygen is used.

2.3.3.2 a series of sump samples are tested.

2.3.4 Carry out the bioassay on the non-aerated demineralized water. This will serve as the analytical blank.

## 3.0 Sample Filtration

3.1 Samples may be encountered which are impossible to clarify following the decantation or centrifuging steps described in the Microtox® Bioassay Method. (Section 3.3.2). Filtration should be considered at this time.

### 3.2 FILTRATION PROCEDURE

3.2.1 The turbid sample should be

3.2.1.1 gravity filtered through a Whatman #1 paper, or

3.2.1.2 vacuum filtered through a 0.45 µ cellulose acetate paper, or

3.2.1.3 filtered through a 0.45 µ cellulose acetate syringe disk, or

3.2.1.4 pressure filtered through a #50 Whatman filter paper.

3.2.2 The filtration apparatus must be clean and free of constituents which could alter the sample toxicity.

3.2.3 pH adjustments, if required, should be carried out prior to filtration.

3.2.4 Perform the Microtox® bioassay on the filtered sample.

### 3.3 QUALITY CONTROL

3.3.1 Test for the presence of toxicants in the filtering medium or apparatus by filtering a small volume of high purity, toxicant free demineralized water through the appropriate filter paper and equipment.

3.3.2 Perform the Microtox® bioassay on the filtered demineralized water.

3.3.3 Carry out the bioassay on the unfiltered demineralized water. This will serve as the analytical "blank".

N.L.C./B.N.C.
3 3286 50162 2886